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Method of making polyamide- polyester- based synthetic fibers

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Detailed Description of the Invention

This invention is related to the method of making the synthetic fiber consisting of the mixed composition of polyamide and polyester.

Objective of this invention is to improve the process operability in the melt spinning of the mixture composition of polyamide – polyester and to provide the fiber of large strength and high Young's modulus.

The fibers which are obtained from the linear polyamide such as poly - ϵ - capro amide (hereinafter, this is abbreviated as nylon 6) and poly hexa methylene adipamide (hereinafter, this is abbreviated as nylon 66) have excellent strength and dyeability and so they are used widely in industry and for apparel. But, on the other hand, they have the shortcomings of low Young's modulus and insufficient dimensional stability.

Because of this, for example when used as the tire reinforcing cord, it is said to cause the drop in tire uniformity and the flat spotting phenomenon and, when used in apparel or carpet, it is difficult to make the fabric or knit product that has the firmness.

On the other hand, the fiber that is obtained from the linear polyester such as poly ethylene tere phthalate (hereinafter, this is abbreviated as PET), has high Young's modulus and good dimensional stability and it can be easily mix-spun with other fibers but there are problems related to the dyeability and the adhesion with other materials.

For this reason, it has been desired to make a synthetic fiber that has the merits of polyamide and polyester and compensates the shortcomings of each other, i.e. has high strength and high Young's modulus, good dimensional stability and rich dyeability and has good adhesion characteristics also. Thus, in the past, various attempts were made. For example, polyamide and polyester were mixed in molten state to a weight ratio of 90:10 ~ 50:50 and, by conducting the spinning and drawing, it was attempted to obtain the fiber that has the characteristics of both fibers and this method (JP No. 26208 - 1965) is well known.

However, polyamide has poor miscibility with polyester and also, between the polyamide and polyester, chemical reaction occurs and so it is difficult to conduct a good melt spinning of the mixture. Furthermore, even if the spinning and drawing were possible, one can not obtain the fiber that has the targeted high strength and high Young's modulus.

In general, the melt spinning of the polymer mixture that is mutually immiscible in the molten state is difficult because of the non-homogeneity of the spinning fluid. The polyamide such as nylon 6, nylon 66 and the polyester such as PET are actually immiscible and so, in the sense that was mentioned above, the melt spinning of the mixture of said polyamide and said polyester requires a technology of a high level.

Another factor that makes the melt spinning of the mixture of polyamide and polyester more complex and difficult is that, between the polyamide and polyester, chemical reaction occurs and that, furthermore, the reaction products give large influences on the spinning process operability and on the physical properties of the fiber that is obtained finally. When the polyamide and polyester are mixed in the molten state (hereinafter, this is sometimes abbreviates as the melt mixing), a block polymer between the polyamide ~ polyester is generated due to the end amino group of the polyamide and, particularly in then case where there is little moisture in the mixed composition, this tendency becomes more pronounced. And, at the time of spinning, such polymer causes continuous increase of the pressure that is applied to the die- filter assembly (hereinafter, this is sometimes abbreviated as the pack pressure) and, in a short time, this causes the spinning to be actually impossible or it causes the melting point depression of the fiber that is obtained finally or generates a weak structure in the fiber, causing the reduction in fiber properties such as the strength.

On the other hand, the end carboxyl group of polyamide promotes the depolymerization of the mixed composite, particularly that of the polyester. Particularly when moisture is present in the mixed composition, this effect is assisted remarkably. For this reason, when the polyamide in which the ratio of carboxyl group among the total end group is large is melt mixed and spun, the yarn non-uniformity is large even if the moisture in mixed composite is kept small and so the yarn breaking increases, making the spinning impossible in some cases; even if the spinning can be conducted, the molecular weight of the fiber that is finally obtained, particularly that of the polyester component in it, becomes very small and the desired fiber of high strength and high Young's modulus can not be obtained.

The present inventors conducted repeated studies to eliminate the above described problems related to the spinning characteristics and fiber physical properties due to the moisture in the mixed composition and due to the said end amino group and the end carboxyl group in the melt mixing of the mixed composition of polyamide and polyester and spinning this to make the fiber and, as the result, found out that, if, as the polyamide component, one uses the polyamide in which the end consists of specific end group and conducts the spinning with moisture contained in the polyamide – polyester mixed composition adjusted immediately prior to the melt mixing, there are no rise of the back pressure or yarn breaking during the spinning and the yarn non-uniformity of the spun fiber becomes very small and so a smooth spinning can be achieved and, in addition, the fiber of very high strength and high Young's modulus can be obtained after the drawing. Thus, this invention was arrived at.

Thus, this invention is characterized as follows: In the case of melt spinning of the mixed chip obtained by mixing the chips of polyamide and polyester to a weight ratio of 90:10 ~ 60:40 or the mixed chip obtained by mixing a small amount of third polymer to this in the molten state, (a) As the said polyamide, one uses the linear polyamide in which the number of end amino group is less than 30 % of the total number of end groups and, also, the number of end carboxyl group is less than 55 % of the total number of end groups and the remaining end group is the inert residual group such as alkyl and allyl, (b) Furthermore, the ratio of the moisture contained in the said mixed chip to the total weight of each polymer component (W wt %) is adjusted to the condition of satisfying the following (1) and (2) simultaneously.

$$W \leq \frac{1}{5} \sqrt{\frac{[NH_2]}{[COOH]}} \quad (1)$$

$$W \leq 0.30 \quad (2)$$

(Here, [NH₂], [COOH] indicate the number of milli equivalents of the end amino group and end carboxyl group, respectively, contained in 1 kg of the polyamide prior to the melt mixing.)

The polyamide that is used in the method of this invention is the nylon 6 and nylon 66 in which the ends are blocked and these polyamides can be made by adding specific amounts of mono carboxylic acid or mono carboxylic acid and mono amine at the time of polymerization initiation or in the middle. Or, to the polyamide in the molten state or in the state where it is dissolved in a suitable solvent, the mono carboxylic acid or the mono carboxylic acid derivative of ester, acid chloride, acid amide can be reacted also. In short, one can use any known optional method that would form the end in which mono carboxylic acid residual group or mono amine residual group is connected by the amide bond in the polyamide. Also, as for the mono carboxylic acid or its derivative or mono amine or its derivative for use in making the end-blocked polyamide that is used in this invention, one that is constructed with the alkyl group or allyl group that would not react in any

form with the amino group, carboxyl group or hydroxyl group or break the amide bond or ester bond or promote the break is preferred. As for the mono carboxylic acid, examples are acetic acid, caproic acid, lauryl acid, benzoic acid and, as for the mono amine, examples are butyl, lauryl amine, stearyl amine.

These polyamides have to be the linear polyamide in which the number of end amino group is less than 30 % of the total number of end groups, number of the end carboxyl group is less than 55 % of the total number of end groups, preferably less than 35 % and also the remaining end groups are the inert residual groups such as alkyl and allyl. Thus, even if the number of end carboxyl group in the polyamide being used is less than 55 %, if the number of end amino group exceeds 30 % of the total number of groups, the spinability of the melt mixture obtained by the melt mixing with the polyester at the moisture content specified in this invention is good but the pack pressure keeps rising and, within a relatively short time, it exceeds the service limit of the spinning apparatus and spinning becomes actually impossible, bringing serious trouble in the process operability at the melt spinning of an industrial scale. Also, with the fiber that is obtained by drawing the spun fiber, the melting point depression is observable and, in addition, good physical properties are not exhibited and, in particular, the strength is small.

Also, even if the number of end amino groups in the polyamide being used is less than 30 % of the total number of end groups, in the case where the end carboxyl group exceeds 55 % of the total number of end groups, for example in the case of using the polyamide in which the end amino group is blocked by dicarboxylic acid, even by the spinning under the condition that would satisfy the condition of the moisture content specified by this invention, there is much depolymerization of the melt mixture, particularly the polyester component, and the viscosity of the melt blend becomes unstable and, as the result, the yarn non-uniformity at the spinning increases and, in the worst case, yarn break occurs frequently, making the spinning impossible. Also, the fiber obtained after the drawing is not the one having high strength and high Young's modulus.

On the other hand, the linear polyester that is used in this invention is the PET or the copolymerized polyethylene tere phthalate in which less than 20m mol % is substituted with other component, glycol component or oxy carboxylic acid.

Also, in the method of this invention, in order to improve the miscibility of polyamide and polyester, a small amount of a third polymer can be added to the said two components.

As for this third component in the method of this invention, use of the polyamide which has the benzene nucleus by more than 20 mol % of the total constitutional components in the main chain is particularly notable. As for such polyamide, examples are poly oxylylene adipamide or ϵ - caprolactam – hexa methylene tere phthal amide co-condensation polymer. If the said third component polymer is added to the mixture of polyamide and polyester by 10 ~ 60 wt % of the polyester, better results are obtained in terms of the spinning characteristics and fiber physical properties as was proposed already (Patent Application No. 14298 – 1967). But, in the case of mixing polyamide that is inert to the

polyester mentioned above, i.e. in the mixed composite which has little interaction, in macroscopic sense, between the non-miscible 2 components of polyamide – polyester in the molten state, the effect of addition of the third component polymer is observable particularly notably.

Further, it also goes without saying that the said polyamide or polyester or the third polymer may contain various types of additives for enhancing the use value, i.e. the light and heat stabilizer, antioxidant, etc. without any problems.

In the melt mixing of polyamide and polyester or of the polyamide, polyester and the third component polymer in the method of this invention, if polyester is less than 10 wt %, the Young's modulus of the fiber obtained can not be pulled up to a value that is sufficiently higher than that of polyamide and so the objective of this invention can not be achieved. Also, when the polyester exceeds 40 wt %, the spinning characteristics goes poor and, at the same time, strength of the fiber drops rapidly, reaching much smaller value compared to that of the polyamide and so, again, objective of this invention can not be achieved.

According to the method of this invention, the moisture content in the mixed chip obtained by mixing the above said components in the form of chips must be adjusted to satisfy the following conditions of (1), (2).

Here, the mixed chip means the mixture of polyamide, polyester and the third component polymer, all of which are in the form of solid polymer. In addition to the so called chip of cylindrical shape, rectangular or plate shape, the chip may contain the pulverized, minute particles also without any problem.

$$W = \frac{1}{5} \sqrt{\frac{[NH_2]}{[COOH]}} \dots\dots\dots (1)$$

$$W \leq 0.30 \dots\dots\dots (2)$$

(In the above equations, W is the moisture content (wt %) with respect to the total weight of polyamide and polyester or with respect to the total weight of polyamide, polyester and the third component polymer) and [NH₂], [COOH] indicate the number of milli equivalents of end amino group and the end carboxyl group, respectively, contained in 1 kg of polyamide prior to the melt mixing.)

Thus, in the case of using the said polymer composition, if the moisture content deviates from the said condition, the yarn breaking and yarn non-uniformity at the spinning increase as in the case of using the polyamide containing large amount of end carboxyl group and, in most cases, spinning becomes practically impossible.

In the method of this invention, the composition that satisfies the above said conditions can be melt-mixed and spun by the common method. As for the apparatus that is used in the melt mixing and spinning, it is preferred to use the extruder type spinning apparatus that is commonly used in the melt spinning of polyamide for melt mixing and spinning but, as long as each component is mixed sufficiently homogeneously, one can use other known, optional melt mixing and spinning apparatus.

By having the spun product further drawn and heat treated by the common method, the fiber of greater practical utility having high strength, high Young's modulus, good dimensional stability, rich dyeability and excellent adhesion characteristics is formed.

In the following, the invention is explained in further detail by the examples of application but these examples of application do not limit the scope of this invention in any way. In the examples of application, [part] means the weight parts. Also, $[\eta]_{mc}$ and $[\eta]_{ocp}$ indicate, respectively, the intrinsic viscosity of polyamide at 35 deg C when dissolved in the refined meta cresol and the intrinsic viscosity of the polyester or the third component polymer at 35 deg C when dissolved in the refined ortho chloro phenol.

Example of Application 1

Directly to the ϵ -caprolactam, the prescribed amount of acetic acid was added and the polymerization was conducted by the common method. After this, by de-monomerizing and drying, the unblocked nylon 6 (nylon a) and 2 types of nylon 6 (nylon b, c) having acetyl group at the end with different concentration of the said acetyl group were prepared. For these, the intrinsic viscosity $[\eta]_{mc}$ and the end group concentration, etc. are shown at the upper part of Table 1.

Next, to 75 parts of each of these nylon chips, respectively, 25 parts of the PET chip of $[\eta]_{ocp} = 0.65$ was added and, by mixing this sufficiently homogeneously by using the V-shaped blender, 3 types of mixed chips (Sample A, B, C) having a moisture content of about 0.13 wt % were prepared.

These samples were spun by using the extruder type melt spinning apparatus of cylinder diameter of 65 mm and comprising the filter section made of metal net and sand and the die section, under the condition of spinning temperature 285 deg C, and melt mixing time of 5 minutes and then, by drawing the spun fiber by using the hot plate of 190 deg C, the 1260 denier fiber consisting of 204 filaments was obtained. The yarn quality of this undrawn yarn at this time is shown at the lower part of Table 1. In this case, in the case of spinning the mixed chip (sample A) using nylon a, the pack pressure continuously rose with the elapsing of spinning time and, after several hours, spinning had to be stopped. The data related to the undrawn yarn and drawn yarn are those related to the fiber which was spun out until then. Also, in the case of spinning the mixed chip (sample C) by using the nylon c, drip occurred frequently and the spinning was barely possible but the drawing could not be conducted smoothly.

As to the mixed chip (sample B) for which nylon b was used, neither the rise of pack pressure nor the drip was observable and so the smooth spinning could be conducted stably. For each of the samples in this example, the moisture content that satisfied the said Equations (1), (2) is shown in Table 1 together with other data.

In this example, the sample that satisfied all of the specifications of this invention regarding the ratio of the number of amino group and number of carboxyl group among the total number of end groups in nylon and the moisture content of mixed chip was sample B. Sample A did not satisfy the specification of this invention regarding the ratio of the amino group and the sample C did not satisfy that of the moisture content of mixed chip. Thus, it is seen that, only in the case where all of the conditions in the method of this invention were satisfied, the spinning characteristics was good and finally it was possible to obtain the fiber of high strength and high Young's modulus.

Table

第 1 表

| | | | | | | |
|----|-------------------|-----------------------|--------------------|-----------|-----------|-----------|
| 1 | 混 合 チ ッ プ 試 料 番 号 | | | A | B | C |
| 2 | ナ イ ロ ン 番 号 | | | a | b | c |
| 3 | 極 限 粘 度 | | de/g | 1.5 5 | 1.5 4 | 1.5 0 |
| 4 | ナ イ ロ ン | アミノ基濃度 | ミリ当量/kg | 4 0.5 | 2 3.5 | 1 0.2 |
| 5 | | カルボキシル基濃度 | " | 3 9.6 | 4 1.5 | 4 0.2 |
| 6 | | アセチル基濃度 | " | 0 | 1 6.8 | 3 2.9 |
| 7 | | 全末端基数に占めるアミノ基 数の割合 | % | 5 0.5 | 2 8.8 | 1 2.3 |
| 8 | | 全末端カルボキシル基数の割 合 | " | 4 9.5 | 5 0.7 | 4 8.2 |
| 9 | 混 合 比 | | ナイロン/PET | 7 5 / 2 5 | 7 5 / 2 5 | 7 5 / 2 5 |
| 10 | 本発明に規定する水分率 | | 重量% | ≦0.2 0 2 | ≦0.1 5 1 | ≦0.1 0 1 |
| 11 | 紡糸時の水分率 | | " | 0.1 2 8 | 0.1 5 1 | 0.1 3 1 |
| 12 | 未延伸糸 | U % | % | 2.4 | 2.5 | 3.3 |
| 13 | 延 伸 糸 | 伸 度 | % | 1 4.6 | 1 3.9 | — |
| 14 | | 強 度 | g / de | 7.4 | 8.3 | — |
| 15 | | ヤング率 | kg/mm ² | 5 5.0 | 6 1.0 | — |

Table 1. (Headings)

1. Mixed chip sample No.; 2 ~ 8. Nylon; 2. Nylon No.; 3. Intrinsic viscosity de/g; 4. Amino group concentration, milli equivalents/ kg; 5. Carboxyl group concentration; 6. Acetyl group concentration; 7. Ratio of the amino group among the total number of end groups; 8. Ratio of the total number of end carboxyl groups; 9. Mixing ratio, nylon/ PET; 10. Moisture content specified in the invention, wt %; 11. Moisture content at the time of spinning; 12. Undrawn yarn, U %; 13 ~ 15. Drawn yarn; 13. Elongation, % ;14. Strength, g/de; 15. Young's modulus, kg/ mm²;

Example of Application 2

In Example of Application 1, the mixed chip samples B, C were vacuum dried at 110-deg C under 0.1 mmHg and the moisture content was reduced to below 0.08 wt %.

These samples were spun by using the same spinning apparatus under the same conditions as in Example of Application 1 and both of the samples B, C exhibited good spinning characteristics. The spun fiber was drawn in the same way as in Example of Application 1. The data at this time are shown in Table 2. Compared to the sample B in Example of Application 1, clearly in this example, the sample C satisfies the moisture content of the mixed chip specified in this invention and, as the result, it is seen that the spinning characteristics was good.

Table

2

第 2 表

| | | | | |
|----|-------------------|----------------------|-----------|-----------|
| 1. | 混 合 チ ッ プ 試 料 番 号 | | B | C |
| 2. | ナ イ ロ ン 番 号 | | b | c |
| 9. | 混 合 比 | N/PET | 7 5 / 2 5 | 7 5 / 2 5 |
| 10 | 本発明に規定する水分率 | | ≦ 0.1 5 1 | ≦ 0.1 0 1 |
| 11 | 紡糸時の水分率 | | 0.0 7 2 | 0.0 7 8 |
| 12 | 未延伸糸 U% | | 2. 2 | 2. 4 |
| 13 | 伸 度 | % | 1 3. 4 | 1 2. 1 |
| 14 | 延 伸 糸 強 度 | g / de | 8. 4 | 8. 6 |
| 15 | ヤング率 | kg / mm ² | 6 5 0 | 7 7 0 |

1. Mixed chip sample No.; 2. Nylon No. ; 9. Mixing ratio, N / PET; 10. Moisture content specified in the invention, wt %; 11. Moisture content at the time of spinning; 12. Undrawn yarn, U %; 13 ~ 15. Drawn yarn; 13. Elongation, % ; 14. Strength, g/de; 15. Young's modulus, kg/ mm².

Example of Application 3.

To ϵ -caprolactam, a prescribed amount of sebacic acid and prescribed amounts of acetic acid and lauryl amine were added and polymerization was conducted by the normal method. After this, by de-monomerizing and drying, nylon 6 (nylon d, e, f) having small concentration of the end amino group and different concentrations of end carboxyl group were prepared.

The intrinsic viscosity $[\eta]_{mc}$ and the end group concentration are shown at the upper part of Table 3 together with those of nylon c in Example of Application 1. Also, in nylon e, f, the concentrations of end group originating from lauryl amine could not be directly determined quantitatively. But, with both of nylon e, f, the degree of polymerization was about same as nylon c and so the total end group concentrations in nylon e and f were regarded as equal to the total end group concentration in nylon c.

Table 3

第 3 表

| | | | | | | | |
|---|-------------------|---------------------|---------|-------|-------|-------|-------|
| 1 | 混 合 チ ッ プ 試 料 番 号 | | D | C | E | F | |
| 2 | ナ イ ロ ン 番 号 | | d | c | e | f | |
| 3 | ナ イ ロ ン | 極 限 粘 度 | de / g | 1.5 6 | 1.5 0 | 1.5 1 | 1.4 8 |
| 4 | | ア ミ ノ 基 濃 度 | ミリ当量/kg | 1 1.0 | 1 0.2 | 1 1.5 | 1 2.1 |
| 5 | | カルボキシル基濃度 | " | 6 7.1 | 4 0.2 | 2 6.8 | 1 2.5 |
| 6 | | アセチル基濃度 | " | 0 | 3 2.9 | 3 0.5 | 3 4.0 |
| 7 | | 全末端基に占めるアミノ基 の割合 | " | 1 4.1 | 1 2.3 | 1 3.8 | 1 4.5 |
| 8 | | " カルボキシル基の割合 | % | 8 5.9 | 4 8.2 | 3 2.2 | 1 5.0 |

| | | | | | | | |
|----|-----------------------|------|----------------------|-----------|-----------|-----------|-----------|
| 9 | 混 合 比 | | ナイロン/PET | 7 5 / 2 5 | 7 5 / 2 5 | 7 5 / 2 5 | 7 5 / 2 5 |
| 10 | 本 発 明 に 規 定 す る 水 分 率 | | 重量% | ≤ 0.0 8 1 | ≤ 0.1 0 1 | ≤ 0.1 3 1 | ≤ 0.1 9 7 |
| 11 | 紡 糸 時 の 水 分 率 | | " | 0.0 4 2 | 0.0 4 7 | 0.0 4 8 | 0.0 4 4 |
| 12 | 未 延 伸 糸 U % | | % | 4.3 | 2.2 | 1.7 | 1.5 |
| 13 | 延 伸 糸 | 伸 度 | % | 1 4.3 | 1 3.6 | 1 1.7 | 1 2.8 |
| 14 | | 強 度 | g / de | 7.5 | 8.6 | 9.1 | 9.0 |
| 15 | | ヤング率 | kg / mm ² | 6 0 0 | 7 5 0 | 8 1 0 | 8 3 0 |

1. Mixed chip sample No.; 2 ~ 8. Nylon; 2. Nylon No.; 3. Intrinsic viscosity de/g; 4. Amino group concentration, milli equivalents/ kg; 5. Carboxyl group concentration; 6. Acetyl group concentration; 7. Ratio of the amino group among the total number of end groups; 8. Ratio of the total number of end carboxyl groups; 9. Mixing ratio, nylon/ PET; 10. Moisture content specified in the invention, wt %; 11. Moisture content at the time of

spinning; 12. Undrawn yarn, U %; 13 ~ 15. Drawn yarn; 13. Elongation, % ;14. Strength, g/de; 15. Young's modulus, kg/ mm²;

Next, to 75 parts of these nylon chips, respectively, 25 parts of the PET chip of $[\eta]_{ocp} = 0.65$ was added, by using a V-shaped blender, thoroughly uniform mixing was done and then the drying was done to prepare 4 types of mixed chips (samples D, C, E, F) having moisture content of less than 0.05 wt %. These samples were spun and drawn using the same apparatus under the same conditions as in Example of Application 1 to obtain the fiber of 204 filaments, 1260 denier. The data related to the undrawn yarn and drawn yarn are shown at the lower part of Table 3. Also, in this example, the moisture content that should satisfy the previously described equations (1), (2) shown in Table 3; all of the mixed chip samples in this example satisfied this moisture content. Examining Table 3, it is seen that, even when the ratio of amino end group in the total end groups as the amide component is about same, a smaller ratio of carboxyl group in the total end group resulted in smaller yarn non-uniformity at the spinning, greater strength and also greater Young's modulus of the drawn yarn.

Thus, in the cases where the ratio of polyamide component among the total end group exceeded 55 %, the yarn non-uniformity in spinning was large and the physical properties of the fiber obtained finally were not good. Also, in the cases where the ratio of the carboxyl group was less than 35 %, it is seen that particularly good spinning characteristics and physical properties were exhibited.

Example of Application 4

To 70 parts of nylon d, e, f, which were prepared in Example of Application 3, respectively, 30 parts of PET chips of $[\eta]_{ocp} = 0.90$ was mixed thoroughly uniformly to prepare 3 types of mixed chips (sample G, H, I) having a moisture content of about 0.15 wt %.

These samples were spun by using the same apparatus under the same condition as in Example of Application 1. The results are shown in Table 4.

In the case of the mixed chip (sample G) where nylon d was used, spinnability of the spun polymer was poor and spinning was entirely impossible. Also, in the case of the mixed chip (sample H) using nylon e, drip occurred frequently and the process could not reach the drawing.

On the other hand, in the case of the mixed chip (sample I) using nylon f, a smooth spinning was conducted.

In this example, the moisture content that should satisfy the previously described equations (1), (2) is also shown in Table 4. It is seen that, even with the relatively large moisture content as in this example, one can conduct a good spinning when all of the conditions specified in this invention are satisfied.

Table

4

第 4 表

| | | | | | |
|-----|-------------------|----------|--------------|--------------|--------------|
| 1 | 混 合 チ ッ プ 試 料 番 号 | | G | H | I |
| 2 | 使 用 ナ イ ロ ン 番 号 | | d | e | f |
| 9 | 混 合 比 | ナイロン/PET | 70 / 30 | 70 / 30 | 70 / 30 |
| 10 | 本発明に規定する水分率 | 重量% | ≤ 0.081 | ≤ 0.130 | ≤ 0.197 |
| 11 | 紡糸時の水分率 | | 0.149 | 0.153 | 0.158 |
| 11' | 紡糸調子 | | 紡糸不能 | ドリツプ発生 | 良好 |
| 12 | 未延伸糸 U % | | — | 4.5 | 2.8 |

1. Mixed chip sample No.; 2. Nylon No.; 9. Mixing ratio, nylon/ PET; 10. Moisture content specified in the invention, wt %; 11. Moisture content at the time of spinning; 11'. Conditions at spinning; 12. Undrawn yarn, U %;

Row 11'.

G. Spinning impossible; H. Drip occurred; I. Good

Example of Application 5

To ϵ -caprolactam, a prescribed amount of lauric acid was added and polymerization was conducted by the normal method and, by de-monomerizing and drying, nylon 6 chip of $[\eta]_{mc} = 1.42$, $[\text{NH}_2] = 12.7$ milli equivalents/ kg, $[\text{COOH}] = 44.2$ milli equivalents. kg ws prepared. The unblocked nylon 6 of the same degree of polymerization had $[\text{NH}_2] = 46.3$ milli equivalents/ kg and $[\text{COOH}] = 45.4$ milli equivalents kg. Therefore, the ratios of amino group and carboxyl group in the total end groups of the said nylon 6 that blocked with lauric acid were calculated to be 13.9 % and 48.2 %, respectively.

To 80 parts of this chip, 20 parts of the PET of $[\eta]_{ocp} = 0.90$ was added and mixed to thorough uniformity to obtain the 2-component mixed chip (sample J). To the said mixed chip was added 5 parts of the chips of ϵ - caprolacctam – hexa methylene tere phthal amide co-condensation polymer of $[\eta]_{ocp} = 0.72$, melting point of 250 deg C, and having a 27 mol % ratio of the benzene nucleus relative to the total constitution components and this was mixed to thorough uniformity to obtain the third component chip (sample K). These were dried to a moisture content of below 0.95 wt %.

These samples were spun and drawn by using the same apparatus and under the same condition as in Example of Application 1 to obtain the fibers. The data related to the undrawn yarn and drawn yarn in this case are shown in Table 5.

The moisture content that should satisfy the previously described equations (1), (2) in this example are shown in Table 5. Moisture content of all of the mixed chips in this example satisfied this condition.

From Table 5, it is seen that, by the addition of the third component polymer, the poor-ness of miscibility of nylon 6 and PET was covered up and, as the result, the yarn non-uniformity was reduced and both of the strength and modulus were improved.

Table

第 5 表

5

| 1 | 混 合 チ ッ プ 試 料 番 号 | | J | K |
|----|-------------------|-----------------------|--------------|--------------|
| | ブレンド比 | ナイロン/PET/ 第3成分ポリマー | 80/20/0 | 80/20/5 |
| 10 | 本発明に規定する水分率 | 重量% | ≤ 0.107 | ≤ 0.107 |
| 11 | 紡糸時の水分率 | " | 0.043 | 0.047 |
| 12 | 未延伸糸 U% | % | 1.2 | 0.8 |
| 13 | 伸 度 | % | 13.2 | 12.0 |
| 14 | 延 伸 糸 強 度 | g/de | 9.3 | 9.7 |
| 15 | ヤング率 | kg/mm ² | 660 | 700 |

1.Mixed chip sample No.; 1B. Blend ratio, nylon/ PET/ third component polymer; 10. Moisture content specified in the invention, wt %; 11. Moisture content at the time of spinning; 12. Undrawn yarn, U %; 13 ~ 15. Drawn yarn; 13. Elongation, % ;14. Strength, g/de; 15. Young's modulus, kg/ mm2;

Comparative Example

The 3-component mixed chip (sample K) which was prepared in Example of Application 5 had a moisture content of 0.138 wt % prior to the drying and this was directly spun by the same apparatus and under the same condition as in Example of Application 1 and the spinning was not possible because of many drips.

As shown in Table 5, the moisture content that satisfies the above equations (1), (2) in this example is below 0.107 wt % and the end group concentration of nylon component satisfies the condition that is specified in the method of this invention and, also, the miscibility of nylon 6 and PET is covered. Thus, it is seen that, even in such a case, a smooth spinning can not be conducted when the moisture content of the mixed chip does not satisfy the above equations (1), (2).

Claims of the Patent

Method of making polyamide – polyester – based synthetic fiber, the method being characterized as follows:

In the process of melt- mixing the mixed chip obtained by mixing polyamide and polyester by the ratio of 90:10 ~ 10:60 in the chip form, or the mixed chip obtained by adding a small amount of a third polymer to this, and melt spinning this,

- (a) As for the said polyamide, one uses the linear polyamide in which the number of end amino group is less than 30 % of the total number of end groups and then number of end carboxyl group is less than 55 % of the total number of end groups and the remaining end groups are the inert residual groups such as alkyl, allyl.
- (b) Furthermore, the ratio (W wt %) of the moisture content contained in the said mixed chip with respect to the total weight of all polymer components is adjusted to the condition that satisfies the following equations (1), (2) simultaneously.

$$W \leq \frac{1}{5} \sqrt{\frac{[NH_2]}{[COOH]}} \dots\dots\dots (1)$$

$$W \leq 0.30 \dots\dots\dots (2)$$

(where, [NH₂], [COOH] indicate the number of milli equivalents of the end amino group and the end carboxyl group, respectively, contained in 1 kg of polyamide prior to the mixing).